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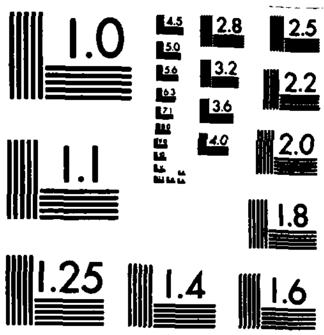

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OBSERVATIONS OF THE USE OF THE SETAPOINT DETECTOR

by

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ABSTRACT

In an attempt to find the reason for certain abnormal test results obtained using the Setapoint detector, a review of experimental results was conducted. Visual observations made during the tests were considered in conjunction with the stop flow and flow points. A number of distinct modes of behaviour, some leading to aberrant results, were identified. The instrument appeared best suited to specification aviation fuels, and middle distillates that produced fine homogeneous wax deposits during the test.

RÉSUMÉ

On a revu les données expérimentales obtenues lors d'essais effectués avec le détecteur Setapoint, afin de déterminer pourquoi certains des résultats s'écartaient des valeurs prévues. Les observations à l'oeil nu faites durant les essais ont été considérées relativement au point d'écoulement nul et au point d'écoulement. On a identifié un certain nombre de modes distincts de comportement dont certains donnaient des résultats aberrants. L'instrument semblait convenir le plus aux carburants d'aviation conformes aux normes et aux distillats moyens produisant de fins dépôts paraffineux homogènes au cours de l'essai.

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1.0.0 INTRODUCTION

In a previous investigation (1), a variety of tests were used to study the low temperature flow behaviour of aviation turbine fuels. These included both specification fuels, and others that had higher than usual freeze points, but whose use might become necessary in the future.

One procedure employed the Setapoint detector, manufactured by Stanhope Seta Ltd. This device measures the resistance to passage of a fuel pumped back and forth across a screen during a programmed cooling regime. In the course of this work a number of irregularities were observed, which suggest limitations on the applicability of the method with some middle distillates, particularly in the presence of added flow improvers.

Drawing mainly on results obtained with an experimental fuel ERBS[1], and with synthetic blends of isooctane and added n-paraffins, a number of types of these irregular phenomena are described.

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1 Experimental Referee Broadened Specification Fuel, proposed by NASA as a reference fuel for engine studies, and possessing an extended distillation range and elevated freeze point.

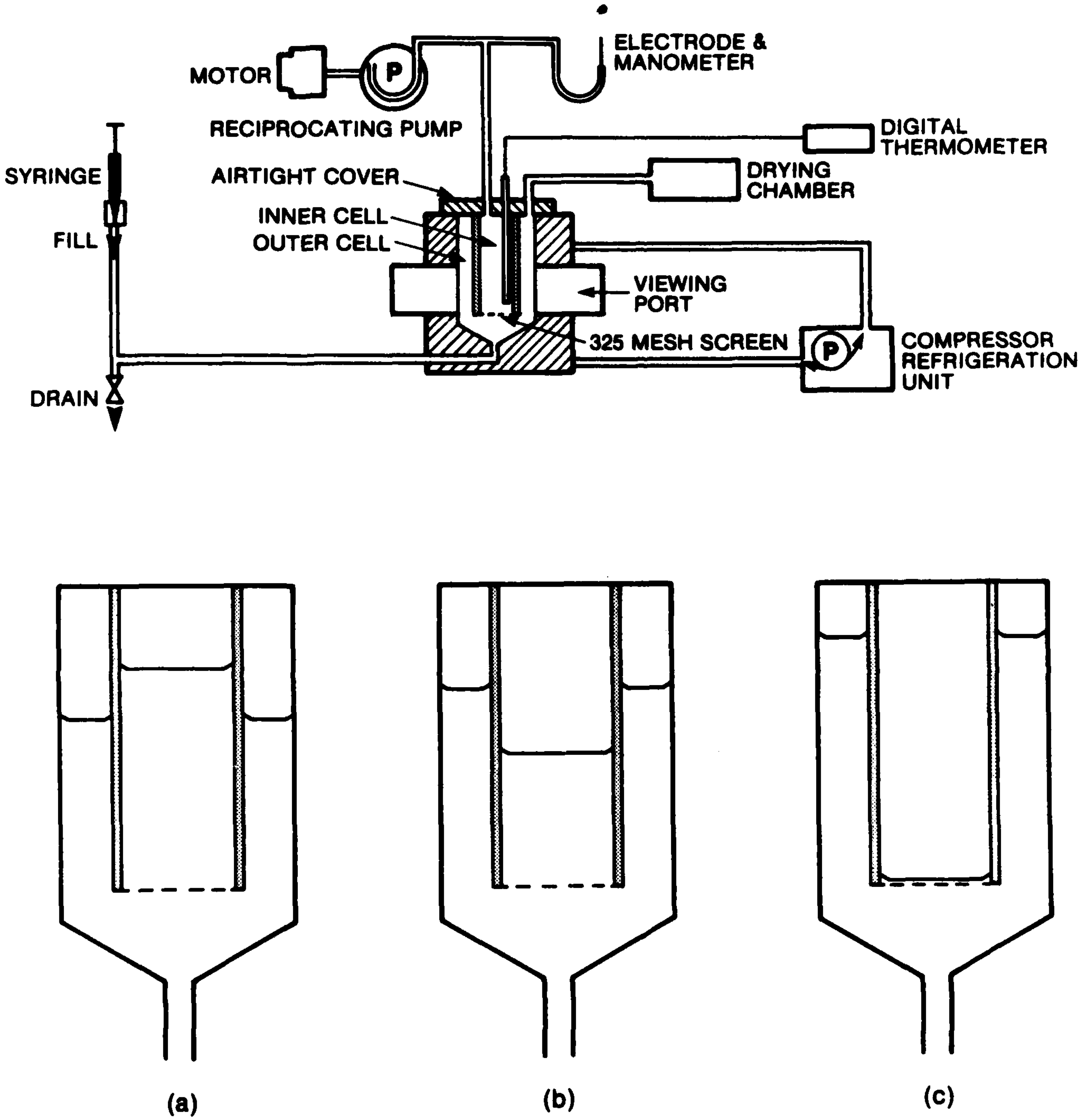

FIGURE 1. SCHEMATIC OF THE SETAPOINT DETECTOR, WITH ENLARGEMENTS OF THE TEST CELL

A. INITIAL FUEL LEVEL IN INNER CYLINDER HIGH
B. INITIAL FUEL LEVEL IN INNER CYLINDER LOW
C. FUEL EXPELLED FROM INNER CYLINDER

2.0.0 EXPERIMENTAL

2.1.0 Setapoint

The apparatus is depicted in Fig. 1. The test cell (shown in the enlargement, 1(a)) consists of a transparent plastic cylinder which contains a concentrically located inner cylinder communicating with the outer one through a 325 mesh metal screen. The sample is injected into the test cell and the cooling programme initiated, while air pulses are applied at regular intervals (20/min) to the fuel in the inner cylinder. These pulses force the fuel periodically through the screen into the outer cylinder, from which it flows back when the pressure impulse is removed. Resistance to passage of fuel through the screen, due primarily to wax formation, is transmitted as a pressure difference to a mercury manometer. When a pressure of one cm mercury is developed and sustained for one second, flow is considered blocked for test purposes, and the apparatus temperature display is arrested at the "stop flow" point. The temperature at which, during rewarming, this pressure-duration criterion is no longer met, is called the "flow" or "resume flow" point.

Temperature settings in the two-stage refrigeration unit are adjusted so that a suitable cooling rate is attained as the fuel approaches the stop flow point. For best results a fresh sample is used for each test to avoid thermal history effects. The flow point has been proposed as an alternative to the ASTM D 2386 test for the freeze point of aviation fuels, and it has been suggested that a correlation exists between the stop flow point and the cold filter plugging test (I.P. 309) for middle distillates.

A viewing port permits observation of the sample as it is pumped back and forth during the test. For the method to give consistent results with the same material it is evident that wax must separate in the same manner at corresponding stages of cooling in successive runs, so that resistance to fuel passage will develop in repeatable manner. The following section surveys Setapoint results for a number of fuels and fuel-flow improver combinations, with visual observations - where the wax formed, its appearance and motion in the chamber as the fuel passed across the screen, and how this appeared to affect location of the stop and flow points. A number of quite distinct modes of behaviour were observed with the Seta apparatus.

2.1.1 Fuels

A selection of test results reported previously (2) for several fuels and blends are presented below in expanded form (i.e., individual determinations are listed with their average and standard deviation). The materials employed were:

1. ERBS-3, one of the variants of ERBS, described by (Seng) (3).
2. Isooctane, in which had been dissolved n-paraffins in the C_9

to C_{20} range so as to duplicate the wax distribution of ERBS 3, called Isooctane I here (2).

3 Isooctane containing 10% by weight of n-paraffins from C_{12} to C_{19}, in the weight ratio 1:2:4:6:6:4:2:1, i.e., peaking sharply at C_{15} - C_{16}) (Isooctane II).

4. A specification JP 5.

The flow improvers, and other additives tested for their effectiveness in this application, were those employed in the previous work (2).

3.0.0 RESULTS AND OBSERVATIONS

3.1.0 JP 5

The Setapoint apparatus was designed in the first instance for the testing of aviation fuels, as an alternative to the D 2386 freeze point test. The following results, obtained over a period of months with a sample of high flash point aviation kerosene, JP 5, illustrate the repeatability attainable.

Stop flow:	-50.6	-50.2	-50.5	-51.0	-51.3	-50.6	-50.7
Resume flow:	-47.8	-48.3	-48.5	-48.5	-48.3	-47.5	-47.6
	-50.8	-50.7	average	=	-50.7 ± 0.3°C		
	-47.6	-47.9	average	=	-48.0 ± 0.4°C		

Stipulations placed on the distillation range of specification fuels result in a narrow wax molecular weight distribution. This is reflected in a rapid onset of the stop flow point; the fuel passes from fluid to completely immobile in three or four strokes of the pump, with the first appearance of a small amount of crystalline wax, the bulk of the fluid remaining clear. Similarly satisfactory behaviour was observed with Jet A-1, the commercial aviation fuel.

3.1.1 ERBS 3

Over the space of several months, six sets of Setapoint determinations were made on the original ERBS without additives. The sets fell into two classes, of which the following are examples.

Trial 1.	Stop Flow:	-32.7	-32.6	-31.7	-30.6	-32.0
	Resume Flow:	missed	-28.1	-26.7	-24.7	-26.6
		-31.2	-30.8	average:	-31.7	± 0.8°C
		missed	-24.5	average:	-26.3	± 1.4°C
Trial 2.	Stop Flow:	-34.0	-32.4	-33.8	-32.3	-32.4
				average:	-33.0	± 0.8°C
	Resume Flow:	-33.0	-31.5	-31.4	-31.2	-31.5
				average:	-31.7	± 0.7°C

Visual observations were similar in the two trials. The solution became cloudy, and a fine granular suspension formed. There was a gradual increase in sluggishness and buildup in manometer pressure to the stop flow point. It was concluded at that time that the readings depended critically on precise instrument settings that controlled the cooling rate, and also varied apparently with the manner of injecting the sample into the instrument. Also, the first run of a series, with the instrument freshly turned on and not thermally conditioned, was frequently erratic.

3.1.2 ERBS 3 + 0.1% w/w Antarox CO 430

The observations in 3.1.1 were clarified by runs conducted with ERBS with the surfactant Antarox CO 430.

Stop Flow:	-31.6	-31.2	-30.2	-29.4	-32.1	-28.6	-29.6
Resume Flow:	-26.1	-29.4	-24.0	-23.6	-30.1	-23.6	-24.2

-32.0	-32.6	-32.3	Runs 3,4,6,7	Stop flow average:	-29.5 ± 0.7°C
				Resume flow average:	-23.9 ± 0.3°C
-30.3	-30.3	-25.9	Remainder	Stop flow average:	-32.0 ± 0.5°C
				Resume flow average:	-28.7 ± 2.1°C

Antarox was in fact ineffective as a flow improver (cf results for ERBS-3 above in 3.1.2) but provided the most clear-cut example of an effect noted with a number of ERBS-additive systems. Two entirely different sets of results were obtained, depending on the relative levels of fuels in the inner and outer chambers of the test cell. When, on injection of fuel to begin a test, the initial level of fuel in the inner cylinder was higher than that in the outer, as in runs 3,4,6,7 above (see Fig. 1(a)) the downward displacement or "stroke" of the fuel in the inner cylinder was large, the initial manometer fluctuations were small (1-2 mm) and both stop and resume flow (values underlined in the table above) were higher than in the reverse case observed in the remaining runs, where the initial fuel distribution was as in Fig. 1(b). Here, large manometer fluctuations (5-6 mm) were observed from the beginning, and both Setapoint readings were lower. As is seen from the averages listed in the table above, the difference is 2.5°C for stop flow and 4.8°C for resume flow.

It was noted that even when initial fuel level in the inner compartment was low (Fig. 1(b)) pump pulsations did not result in expulsion of air through the metal screen, but only fuel. Evidently surface tension effects at the air-fuel-screen interface are such that the pressure head was insufficient to push air bubbles through the screen; instead, the resistance to air passage was reflected in the pressure rise noted at the manometer. Thus, differences in manometer behaviour with different fuel distributions can be explained. It is not understood, however, why this should influence Setapoint determinations as greatly as it does. With "well-behaved" fuels such as JP 5, fuel levels, exact instrument settings and previous thermal conditioning of the instrument had no effect on Setapoint results. This was confirmed in the work reported in 3.1.0 by deliberately varying these parameters.

3.1.3 ERBS 3 + 0.1% w/w Paradyne 25

Stop Flow:	-48.1	-48.2	-48.2	-48.2	Average: -48.2 ± 0.1°C
Resume Flow:	-45.0	-45.2	-45.1	-45.2	Average: -45.1 ± 0.1°C

3.1.4 ERBS 3 + 0.1% w/w Paramins ECA 7973

Stop Flow: -47.3 -47.0 -47.8 -48.1 -47.7 -47.8 -47.4 Average: -47.6 ±0.4°C
Resume Flow:-44.6 -45.2 -44.7 -44.7 -44.7 -44.5 -44.6 Average: -44.7 ±0.2°C

The flow improvers in the two trials above are very potent, reducing Setapoints for ERBS 3 by 15-20 degrees. They are apparently equally effective and results for both are satisfactorily repeatable. Visual observation however shows two quite different phenomena occurring. With Paradyne 25, the wax comes out as a milky suspension, filling the field of vision and moving without resistance through the screen. As the temperature falls the suspension becomes heavier until the fuel is nearly opaque, with no evidence of particles or any visibly heterogeneous structure. Pumping continues unimpaired to quite low temperatures· the first increase in viscosity is noted within a degree or two of the stop flow point.

With ECA 7973 a granular deposit forms on the outer walls of the chamber and builds up an adherent white snow-like mass, with only a small amount of detached granules moving in solution. At the stop flow point only a narrow passage is left unblocked in the centre of the test cell, in which a small volume of nearly transparent fluid is pumped across the screen. The close similarity in Setapoint results is in contrast to the very different physical nature of the separated wax.

3.1.5 ERBS 3 + 0.1% w/w Tolad T40

Stop Flow:	-46.6	-45.3	-37.0	-35.0	-45.7	-34.6
Resume Flow:	-41.2	-39.5	-27.8	-28.8	missed	-30.5

-32.9	-38.7	-37.3	-38.0	-36.6	-29.4	-32.9	-33.3
-27.9	-36.5	-31.5	-31.7	missed	missed	-27.0	-27.4

-39.0	-38.5	-31.8	average (17 runs)	-37.2 ± 4.9°C
-29.0	-32.6	-26.9	average (14 runs)	-32.0 ± 5.0°C

The extremely low readings in runs 1,2 and 5 were due to deposition of an adherent deposit on the walls of the cell, as with ECA 7973 noted above. The extreme variability in behaviour, which led to the accumulation of the large number of results reported here, was related in part to the initial distribution of fuel in the cell, as in 3.1.2 above, but also to the nature of the separated material. It appears in this system as flat tablets and stringers which because of their shape settle only slowly out of solution. As a consequence of the pump pulsations they are carried repeatedly up against the bottom of the screen where they may or may not adhere, in intrinsically irreproducible fashion. This led to numerous "false starts" during the rewarming toward resume flow, caused by partial blocking and unblocking of the screen. This behaviour is reflected in the extremely large standard deviations reported.

In one run flow remained completely blocked while the chamber warmed up to −27°C, and the suspended wax almost completely dissolved, this was followed by sudden resumption of vigorous pumping, while a large flat fragment of wax which had apparently been blocking the screen floated into the field of vision.

3.1.6 Isooctane-I

In a set of fourteen runs carried out with this blend, the first four and the last ten behaved in two entirely different ways.

First Mode	Stop Flow:	-33.0	-33.0	-33.2	-33.2	Average: -33.1 ± 0.1°C		
	Resume Flow:	-29.9	-30.4	-31.5	-31.8	Average: -30.9 ± 0.9°C		
Second Mode	Stop Flow:	-35.2	-34.2	-35.3	-36.2	-35.3	-34.5	
	Resume Flow:	-35.2	missed	-35.3	-36.3	-35.5	-34.5	

-36.0	-35.3	-36.6	-34.8	Average:	-35.3 ± 0.8°C
-36.0	-35.3	-36.6	-34.8	Average:	-35.5 ± 0.7°C

All runs were made with the same instrument settings. In the first set the stop flow point was sharp, with only a small amount of crystalline material visible at the end, as with JP 5; in the second set we observed gradual development of a cloudy suspension and increase in viscosity prior to the stop flow point, very much like the ERBS 3 whose wax distribution it reproduces. This was the most notable instance of a phenomena observed with several systems, a series of similar runs followed by an apparent shift in the mode of operation of the instrument, so that quite different sets of results are obtained, all operating conditions being the same.

Isooctane-I treated with a number of the flow improvers found in previous work to be effective with ERBS 3, exhibited the behaviour shown by ERBS 3 with ECA 7973, a wax deposit adhering to the chamber walls.

3.1.7 Isooctane-I + 0.1% w/w Acryloid 154-70

Stop Flow:	-39.3	-39.2	-39.1	-39.4	Average:	-39.6 ± 0.9°C
Resume Flow:	-35.3	-34.4	-35.3	-39.7	Average:	-35.9 ± 2.2°C

In this s tem the originally cloudy granular suspension collected into compa masses which settled out of solution, so that as cooling proceeded the ield of vision became lighter rather than more opaque. As the fuel was pumped back and forth these settled deposits did not approach the screen, in contrast to the wax fragments observed in the ERBS 3 - Tolad T40 system, whose shape helped to keep them in suspension.

3.1.8 Isooctane-II

Stop Flow:	-29.5	-29.5	-29.2	-29.2	-29.4	-29.3*	-29.4*	-29.4
Resume Flow:	-28.0	-27.7	-27.8	-27.1	-27.6	-27.7	-27.5	-27.4

Stop Flow Average: -29.4 ± 0.1°C
Resume Flow Average: -27.6 ± 0.3°C

* Solution seized up in cell without initiating stop flow signal.

With a sharply peaking wax distribution, Isooctane II exhibited in even greater degree than JP 5 the phenomenon of sudden freeze up, fluid motion ceasing and the stop flow signal appearing instantaneously during a single pump stroke. Occasionally seize-up in the fluid occurred with no stop flow signal. The most probable explanation is that if instantaneous holdup occurs at the bottom of the stroke, when all fuel has been expelled through the screen (Fig. 1(c)), the fuel until that moment having been quite fluid, the air space in the inner cylinder may be large enough to absorb succeeding air pulses without reaching the one cm mercury pressure differential needed to trigger the "stop flow" signal.

3.1.9 Isooctane II + 0.1% w/w ECA 7973

Stop Flow:	-33.9	-32.4	-48.3	-36.4
Resume Flow:	-34.4	-33.3	-48.7	-36.8

Isooctane II did not respond to most of the flow improvers used with it (2); comparison of 3.1.8 and 3.1.7 demonstrates this. The one extremely low value reported above is due to precipitation of the wax as an adherent deposit on the walls of the test cell, another instance of the random occurrence of two different modes of wax formation in the middle of a series of runs conducted identically.

4.0.0 SUMMARY AND CONCLUSIONS

The intention of this data review was not to look for correlations between the Setapoint results and those of other test procedures, but rather to examine the method for consistency and repeatability, and to identify the causes of irregular behaviour. The Seta apparatus was clearly best suited in this connection for specification aviation fuels, whose waxes had a relatively narrow molecular weight distribution. It also gave good results with ERBS, with a wide n-paraffin base, when this fuel was treated with an effective flow improver that could produce a finely divided and mobile wax suspension.

Among the phenomena observed were:

a. shifts in instrument behaviour during a series of runs, so that two consistent sets of results differing from each other were recorded, with the same sample and operating conditions.
b. the influence of initial sample injection and distribution in the test cell. In certain cases this was responsible for the two modes of operation noted in (a) However it is not known why this effect is operative only with certain fuels and not, for example, with JP 5.
c. the formation, as in ERBS-3 + Tolad T40 of wax in a physical form which resulted in widely variant Setapoints from run to run.
d. isolated runs as in 3.1.9, in an otherwise consistent series.

One problem is the physical regime the fuel is subjected to in the test. Repeated passage across a fine screen under continuous cooling applied at the walls of the test cell is not relatable to what happens during fuel storage and preparation for combustion, in either jet or diesel engines. Thus, the experience with ERBS treated with Paradyne 25 and with ECA 7973, described in Sections 3.1.3 and 3.1.4, seemed to show the latter flow improver to disadvantage, but the wax would almost certainly not separate in actual use as it does in the test method. In addition the small scale of the test distorts and magnifies the effect of inhomogeneities, as seen, for example, with ERBS + Tolad T40.

It appears that with other than specification aircraft fuels careful visual scrutiny of the wax formation process is essential to avoid misleading results. This kind of examination could be very useful in experimental studies of low temperature phenomena, if the difference between physical treatment of the fuel in the test and in actual use is borne in mind.

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